Materials at the Nanoscale

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Collaborators:
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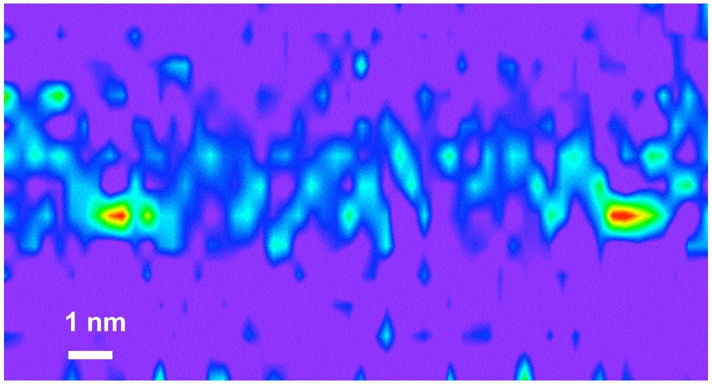
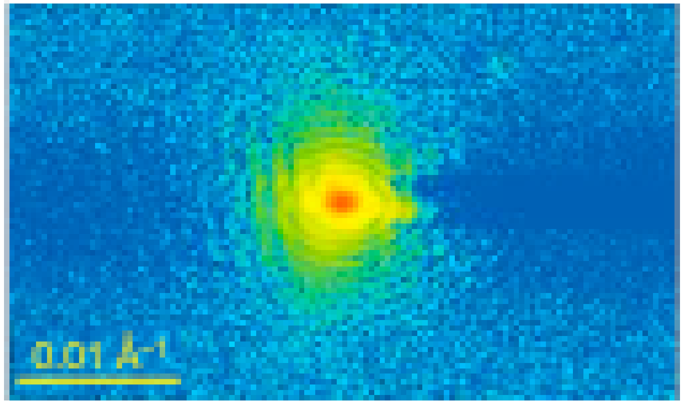
[2]Materials Science Division
[3]X-ray Science Division
Argonne National Laboratory

Outline

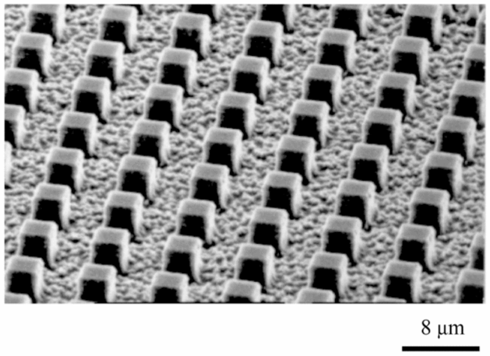
- Materials Science with a Hard X-ray Nanoprobe
- Capabilities for a Hard X-ray Nanoprobe

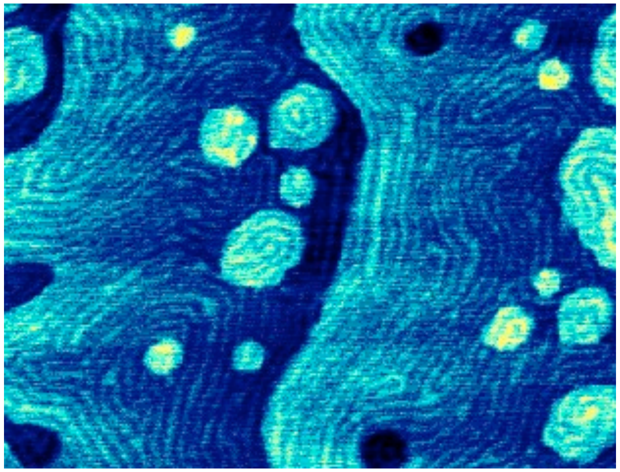




Key Issues in Nanoscale Materials Science

- Fabricated and natural nanostructures
- Emergent properties due to competing interactions: Composition, stress, field, polarization, …
- Non-uniformities and proximity effects from boundary conditions, interfaces, domains
- Synthesis, stability, and dynamics

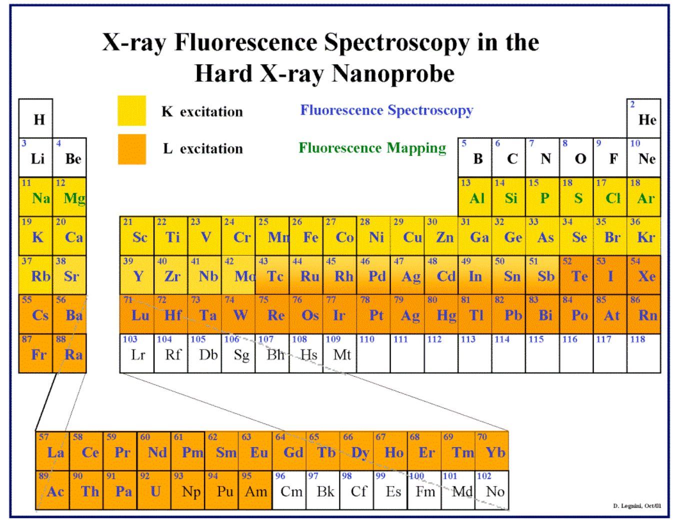


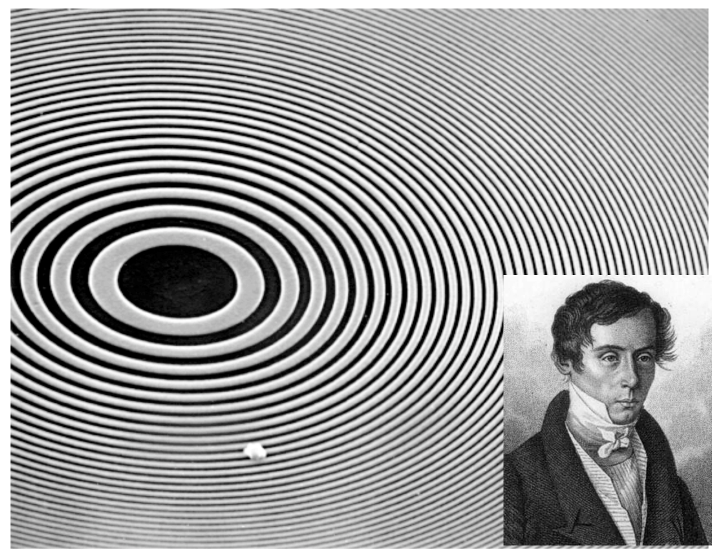


Hard X-ray Nanoprobe Capabilities

- Hard X-rays give large penetration → in situ studies of sample interiors, in environments and fields

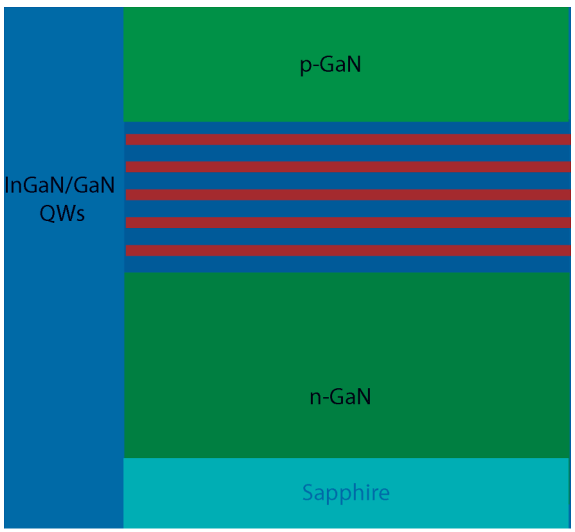
- X-ray nanoprobe capabilities
  - X-ray fluorescence: atto-g elemental sensitivity, chemical state sensitivity
  - Diffraction: sensitivity to crystallographic phase, strain, orientation
  - Coherent x-ray studies: disorder, imaging
  - Magnetic contrast using polarized x-rays
  - Dynamic studies using chopper

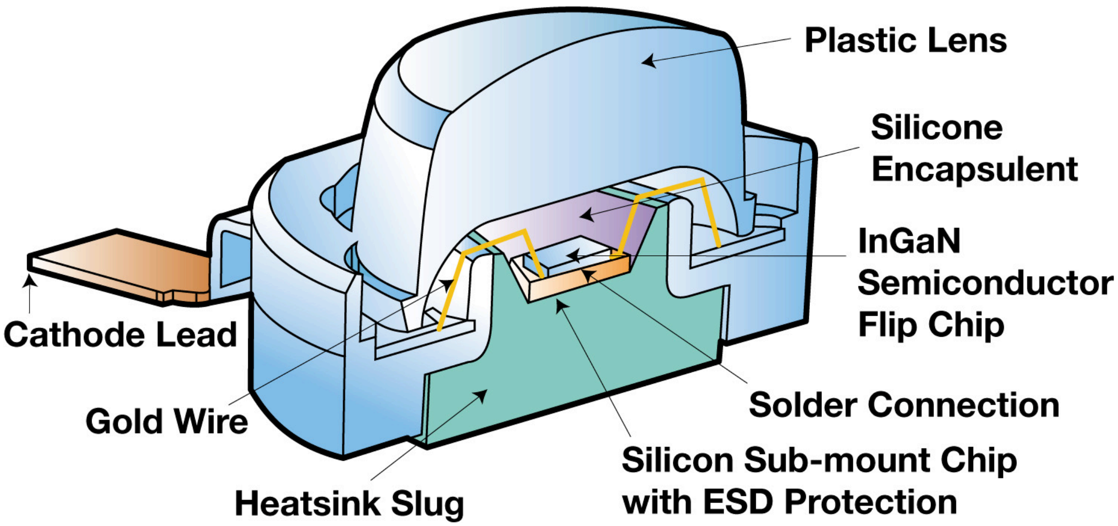




LEDs for High Efficiency Lighting

- Understanding InGaN structure and synthesis has been identified as a key materials issue in the continued improvement in efficiency of LEDs for general illumination

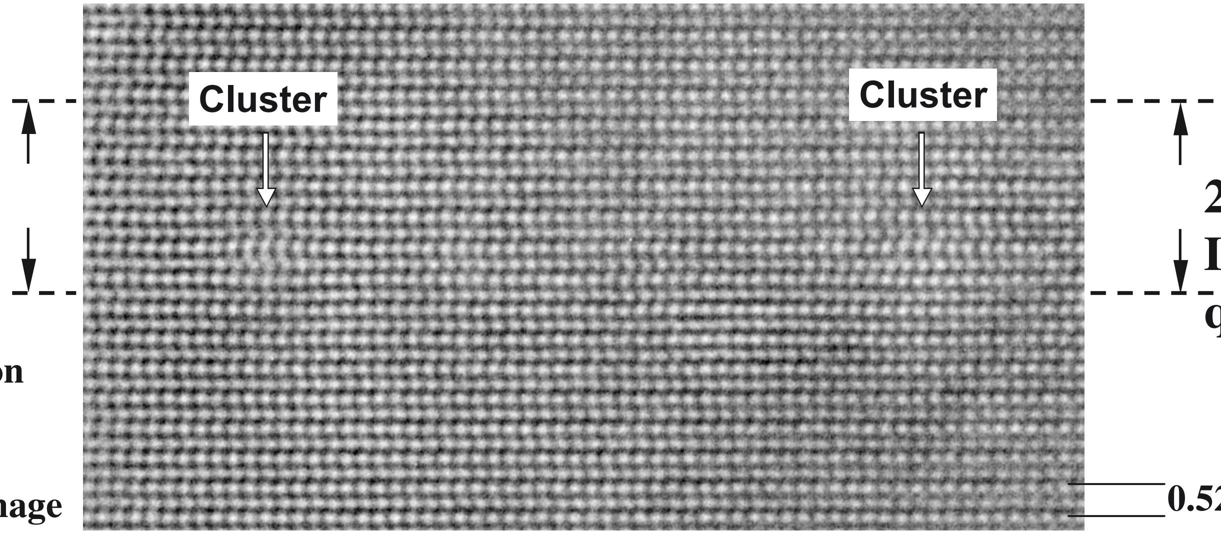




Cross section view of Luxeon Emitter

A. Munkholm (Philips Lumileds) and B. Stephenson (ANL)

Nanoscale Indium Clustering?

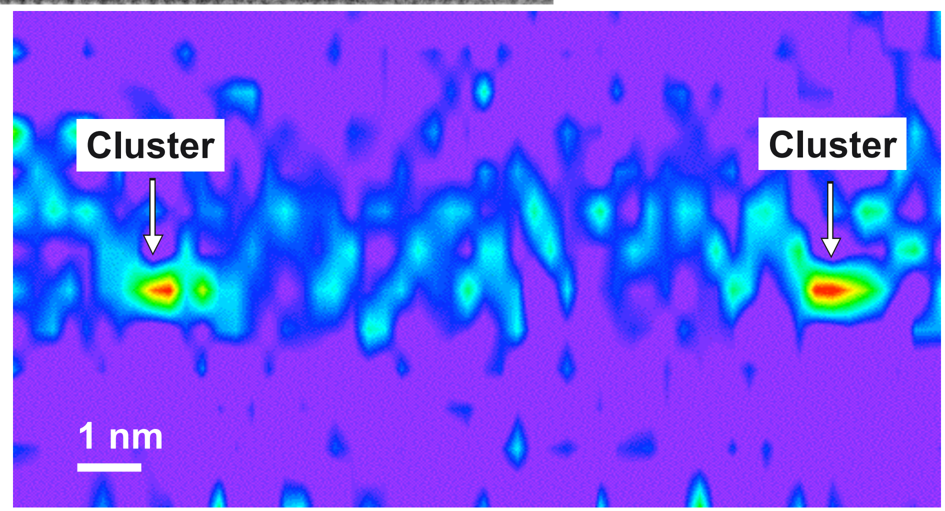


High-resolution Transmission Electron Microscope image

ncem

J.R. Jinschek, Ch. Kisielowski
National Center for Electron Microscopy, LBNL

False-color image emphasizing changes in spacing of atomic columns



X-ray Nanoprobe can determine whether indium clusters are present in as-grown quantum wells

Self-Organized Nanoscale Pattern Formation

Equilibrium domain patterns

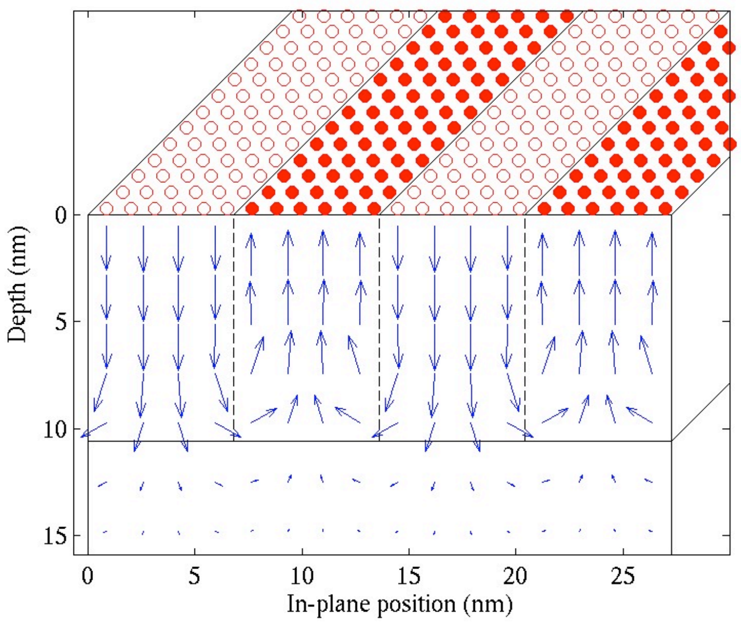
Key scientific areas:

- Understanding competing interactions and energetics that lead to equilibrium non-uniformity, e.g. long-range vs. short-range, ...
- Can we control morphology using e.g. fields, chemistry, templates ... ? How and where do domains form?
- Real-time studies of dynamics of nanoscale domains with buried interfaces, in processing environments

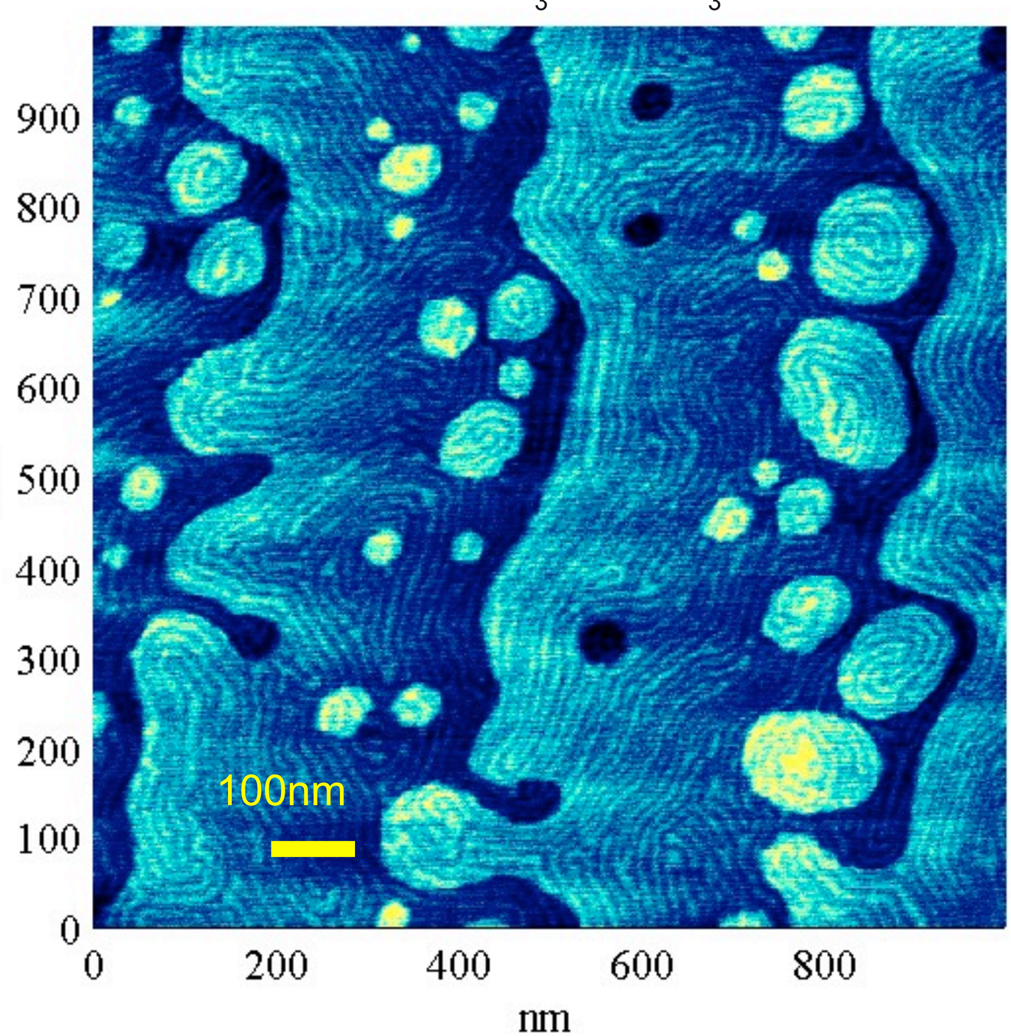
Examples: ferroelectric and ferromagnetic domains

Nanoscale 180° Stripe Domains in Ferroelectrics

- After discovery using x-ray scattering, we have recently been able to image them in AFM
- Domain walls follow surface steps
- Do ions decorate stripes?

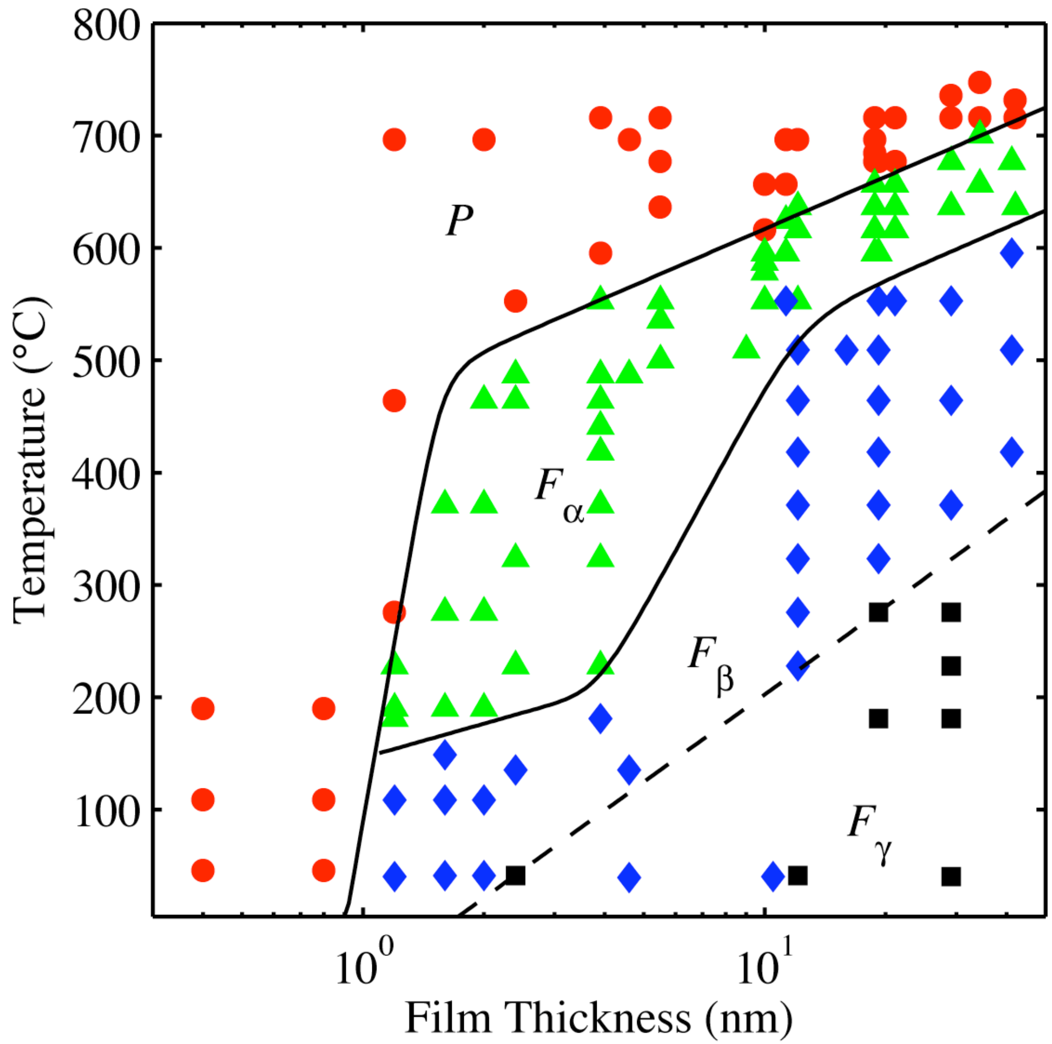


10nm $PbTiO_3$ on $SrTiO_3$



C. Thompson *et al.*

Transitions in Stripes: Temperature and Film Thickness, $PbTiO_3$ / (001) $SrTiO_3$



- Below T_C, see two regions of stripes with different periods
- What determines transitions? Are stripes dynamic?

● *P*: Paraelectric

▲ F_α: Ferroelectric, stripe α-phase

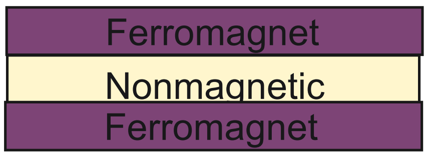
◆ F_β: Ferroelectric, stripe β-phase

■ F_γ: Ferroelectric, no stripe domains

S.K. Streiffer *et al., Phys. Rev. Lett.* **89**, 067601 (2002)

Magnetic Structure of Nanodots

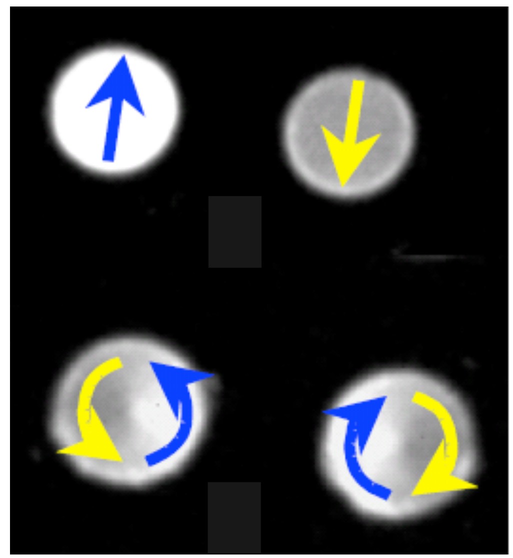
X-ray Magnetic Circular Dichroism to Image Buried Magnetic Layers

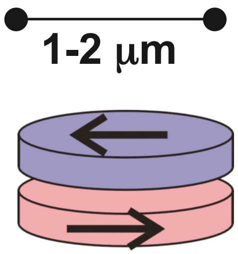


Spin-valve geometry

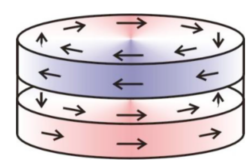
PEEM images (ALS):
Surface Magnetization

Buchanan, Guslienko, Doran, Scholl, Bader and Novosad., *PRB,* **72**, 134415 (2005).





20/20/20 nm
Py/Cu/Py



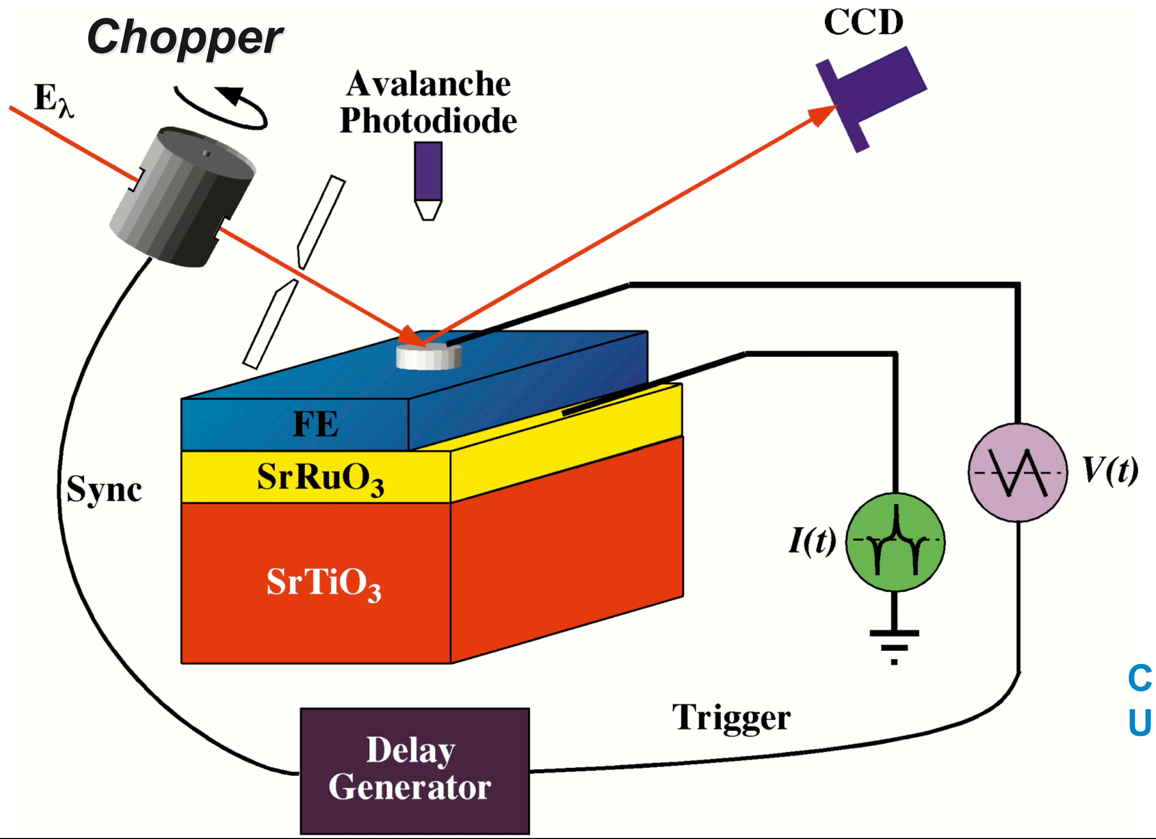
20/1/20 nm

- ***What is the magnetization state of the bottom layer?***

K. Buchanan, V. Novosad, A. Hoffmann, S. D. Bader (ANL),
J. Sort (Univ. Barcelona)

Stroboscopic Observation of High Speed Switching of Nanoscale Ferroelectrics

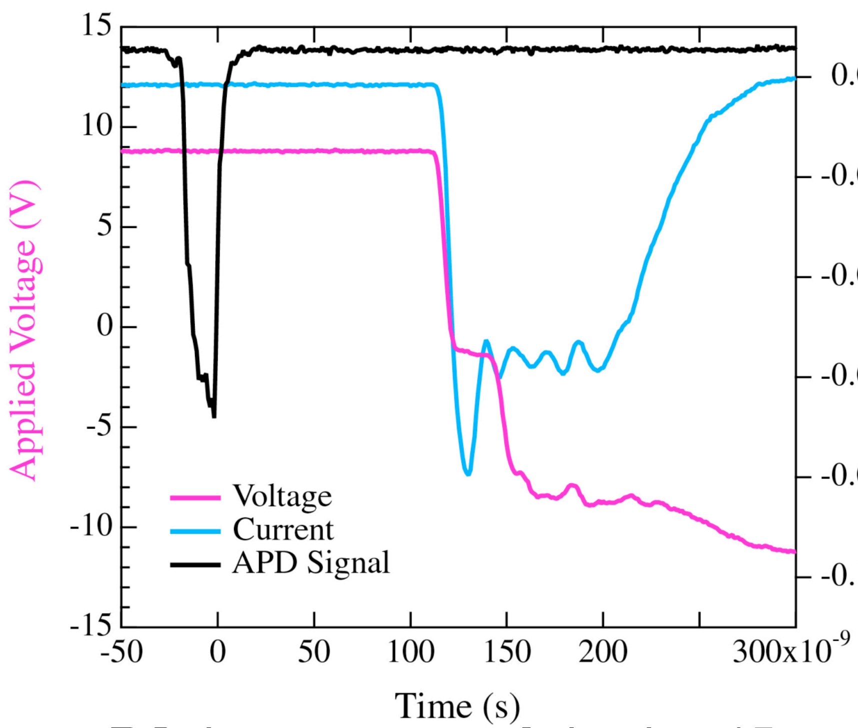
- Cycle voltage on ferroelectric sample in sync with x-ray pulses
- At each point in voltage sequence, observe lattice parameter change due to polarization and piezoelectric effect

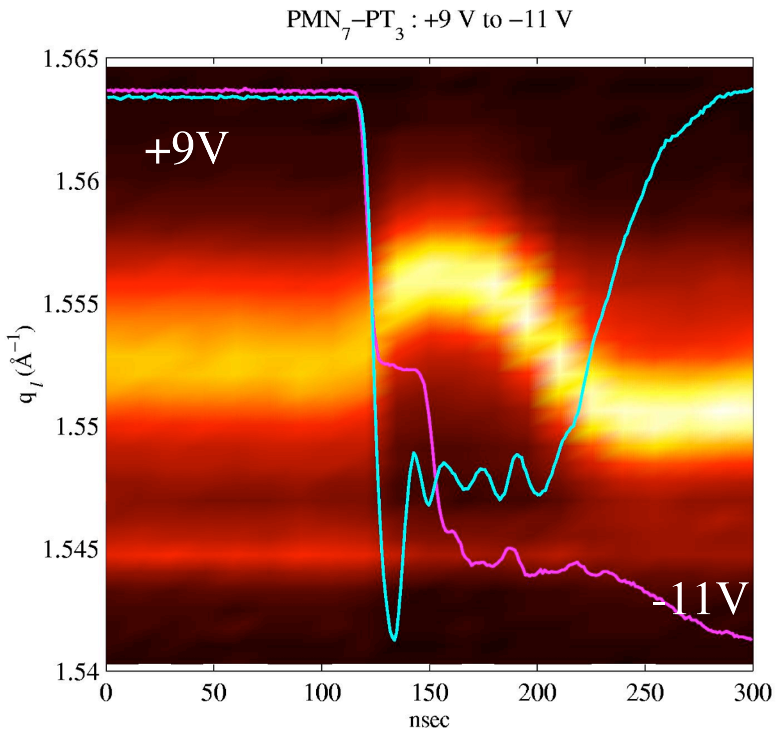


C. Thompson (Northern Illinois Univ.), S. Streiffer (ANL)

Time Resolution Limited by Device Size

PMN_7-PT_3 Response to a Step Voltage

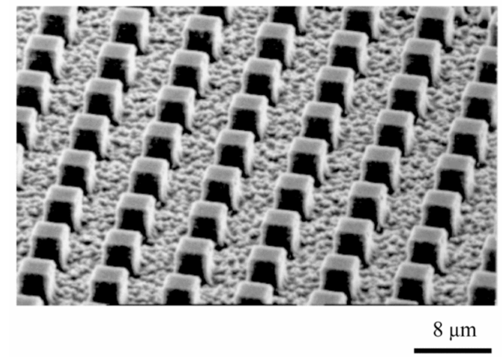


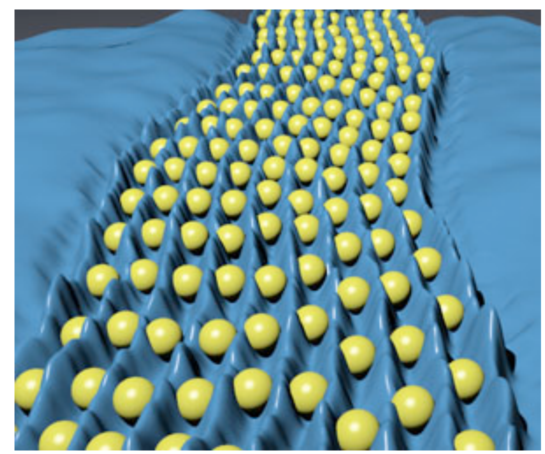


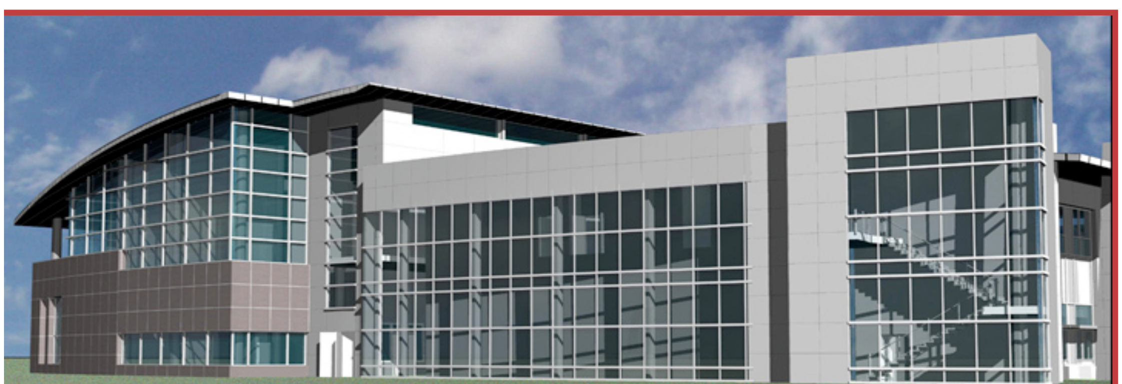
- RC time constant of circuit ~ 15 ns, given current device size (100 um)
- Intrinsic < 1 ns time scales will be accessible in 1 um size

Interaction with Center for Functional Nanomaterials

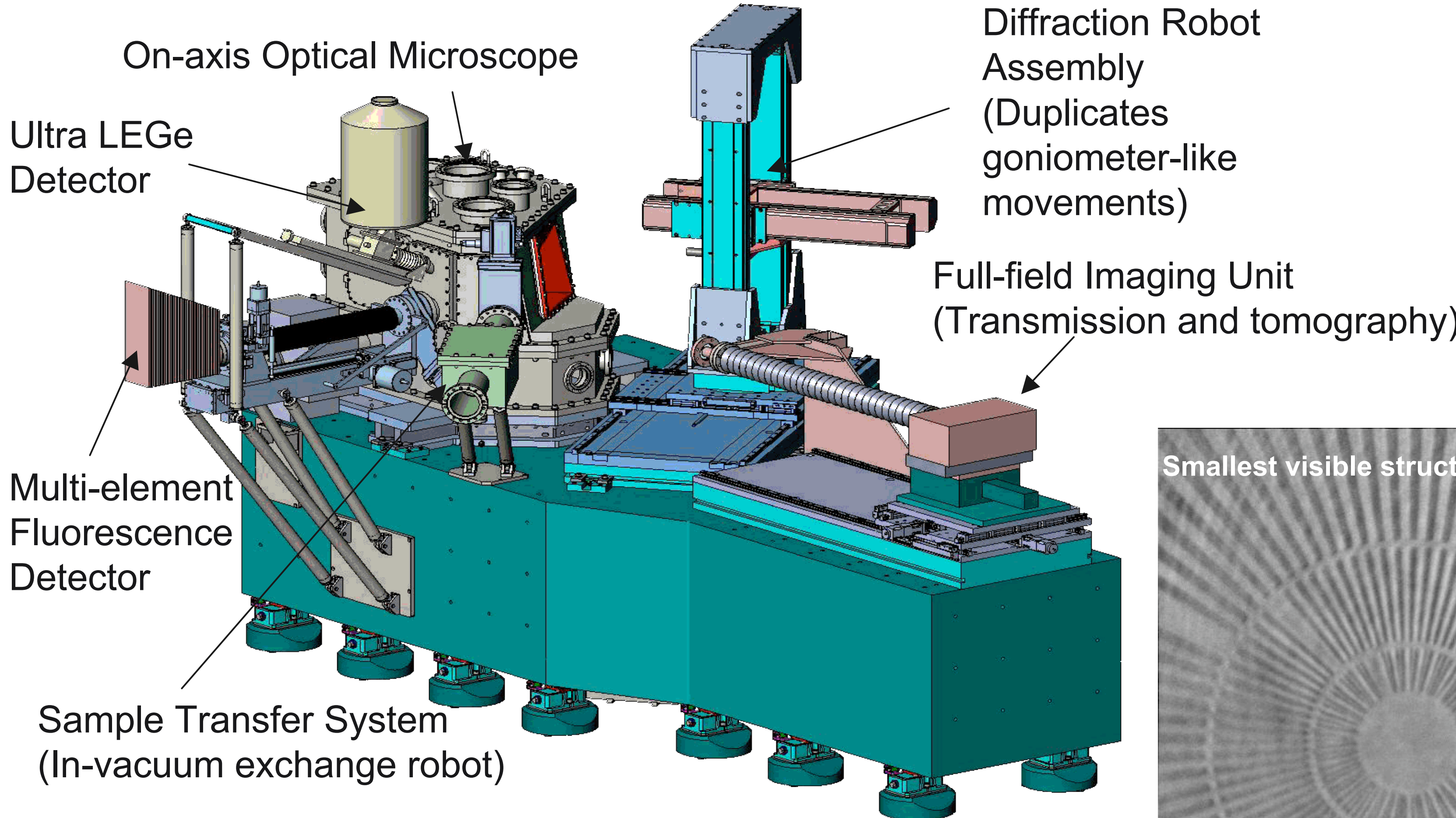
- Nanotechnology - a frontier opened by new tools for analysis and fabrication
- Nanoscience - provides understanding of functionality and synthesis processes
- DOE Nanoscience Research Centers are new user facilities that bring together advanced nanoscale synthesis and analysis facilities
- Both top down (patterning and lithography), bottom up (self assembly), and hybrid approaches
- Expert staff as well as large-scale facilities

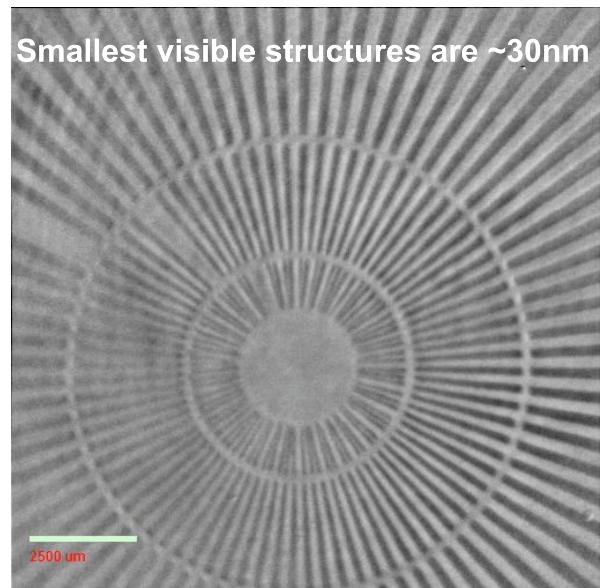




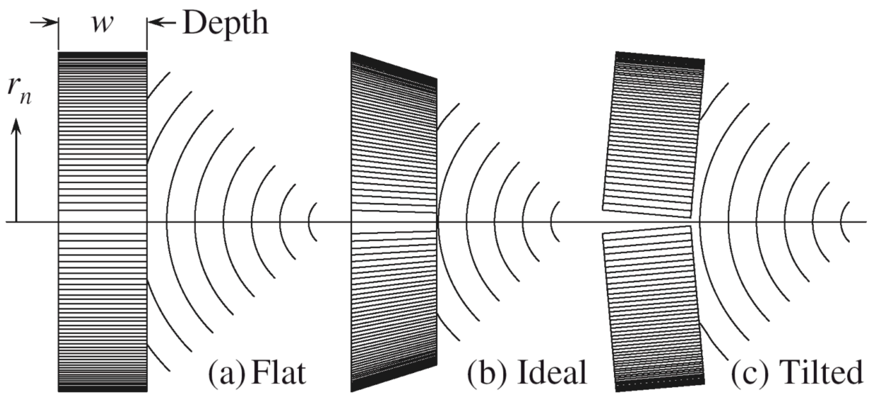


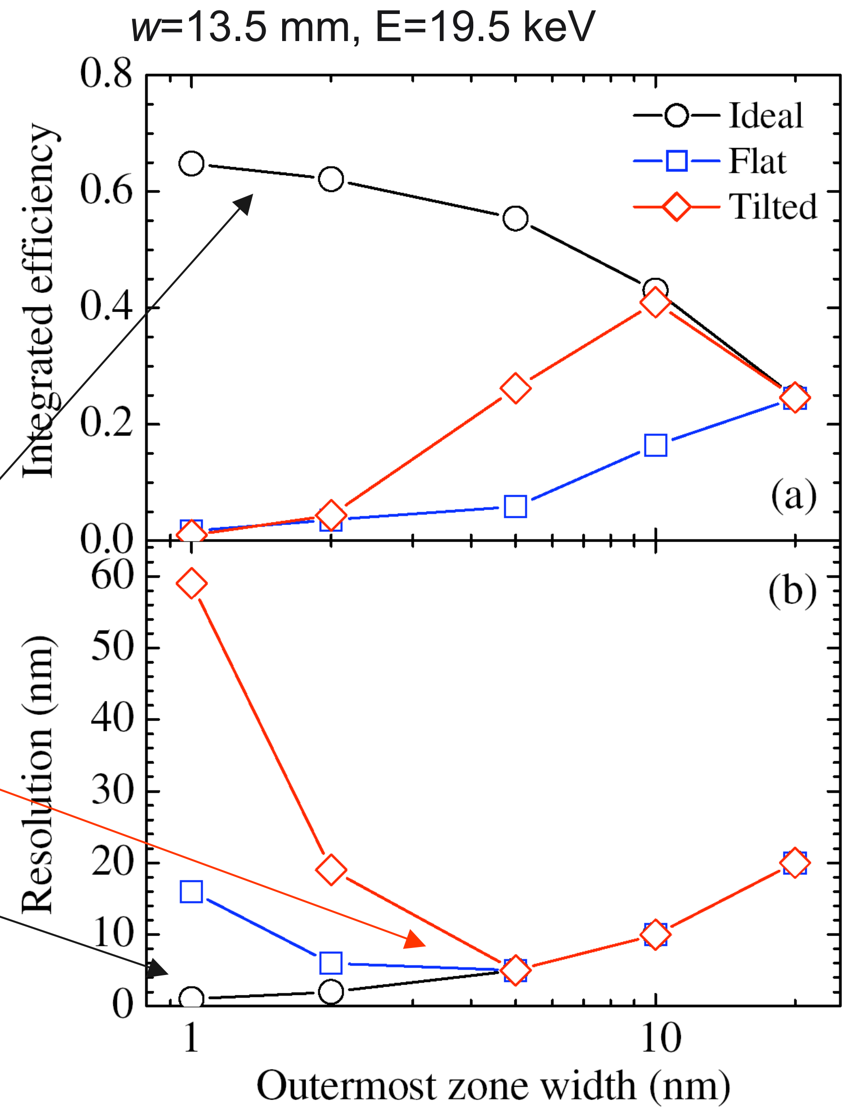
Nanoprobe Instrument at CNM/APS





Ultimate Optics for Nanometer Hard X-ray Focusing: Multilayer Laue Lens

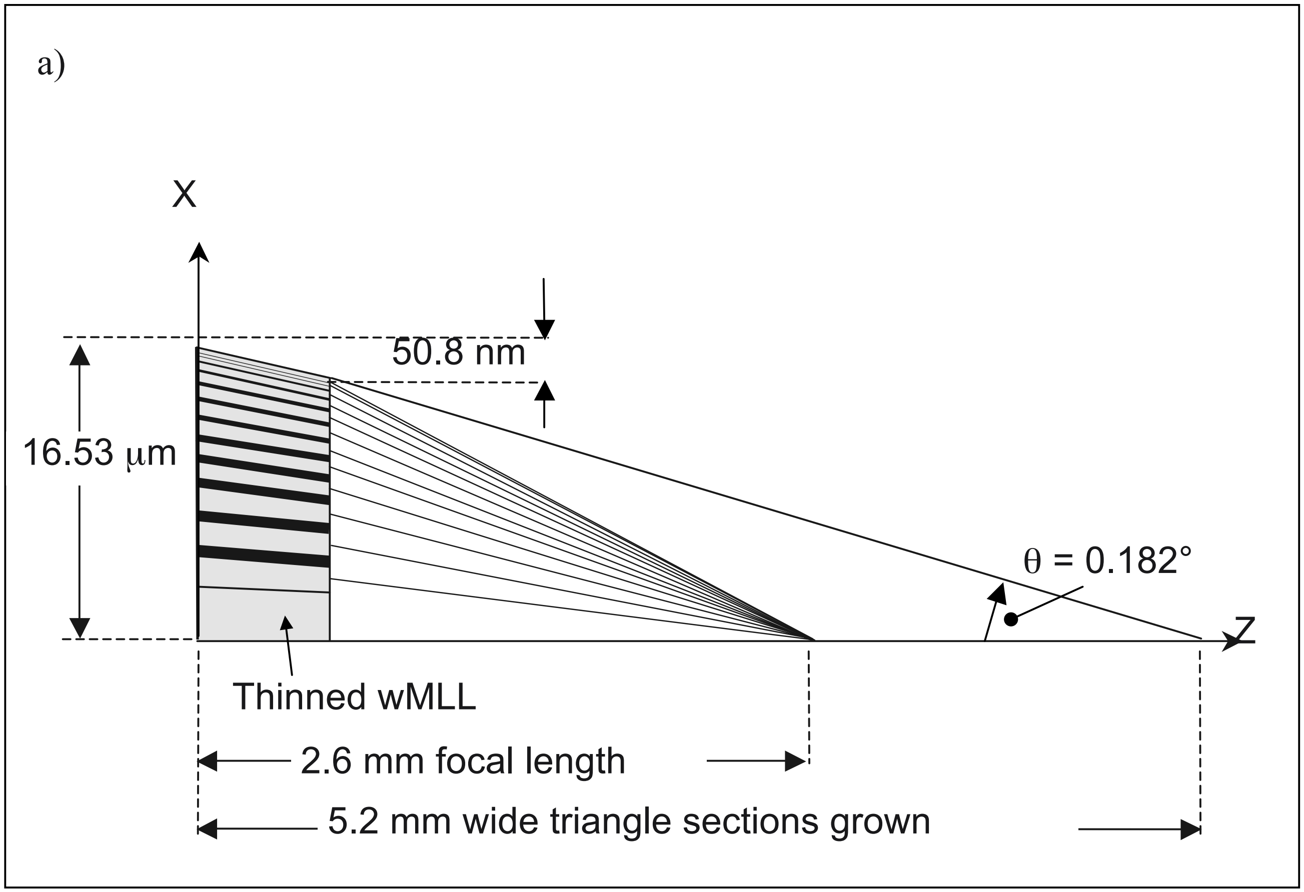




- Tilted MLL structure:
  - 6 nm FWHM feasible
- Ideal "wedged" structure:
  - High diffraction efficiency
  - FWHM below 1 nm feasible

H. C. Kang et al.
Physical Review Letters **96,** 127401 (2006)

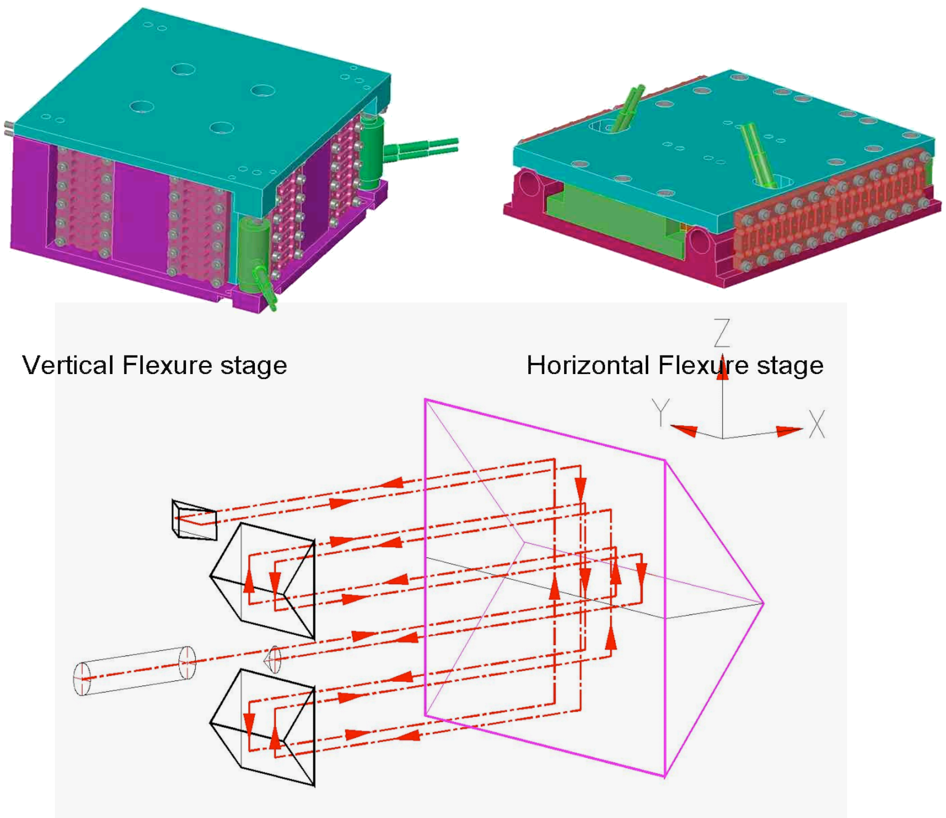
Progress of Deposition of Wedged Multilayer Structures



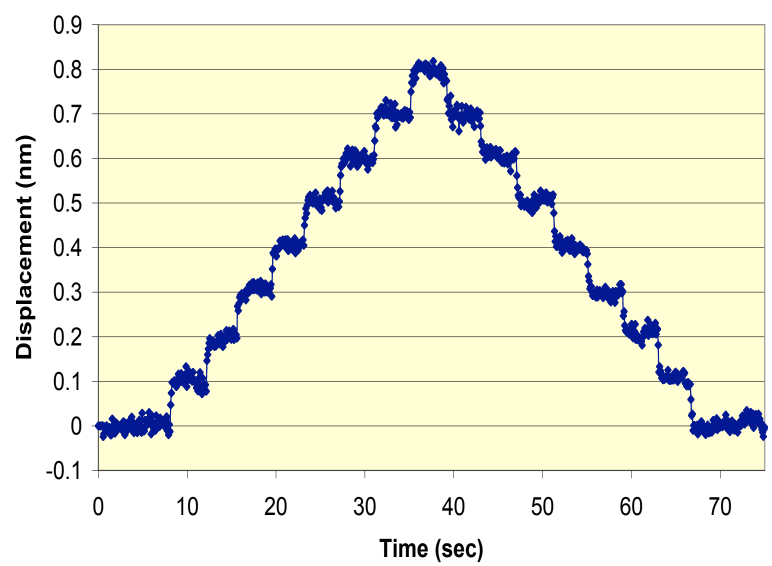
- R. Conley et al. (APS)

Nanoprobe Scanning Design

- Position of sample is interpreted by controls system
- Sample and optics locations are monitored and variations in the sample position and optics are corrected with the optics module
- Motions of the sample and zone plate are “locked” together so measurements are made of the same spot on the sample (kHz)



Response of prototype control system to commands for single Angstom steps:

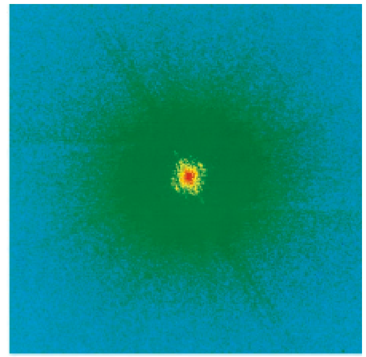


Coherent Diffraction Imaging

Rapid development in inversion algorithms

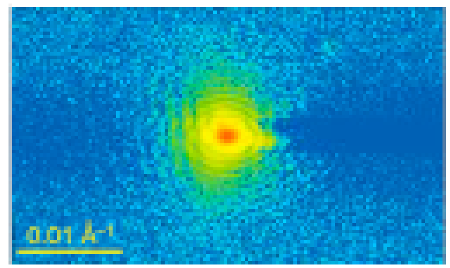
- Hard x-rays: forward scattering

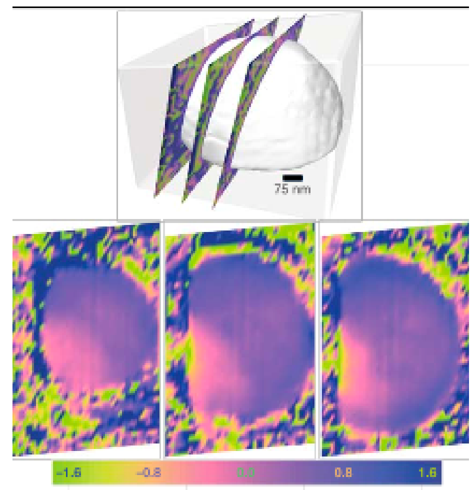
  *J. Miao et al., PNAS **100**, 110 (2003)*



- Hard x-rays: Bragg scattering

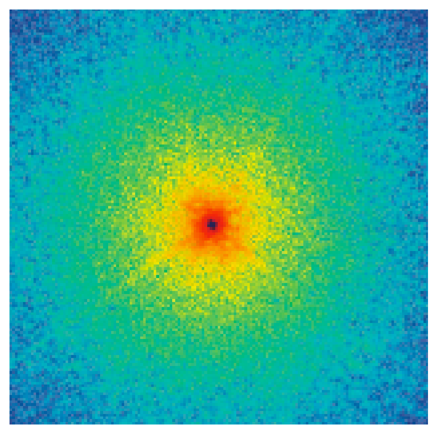
  *M. A. Pfeifer et al. Nature **442**, 63 (2006)*

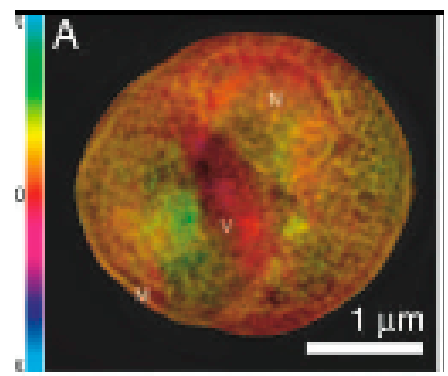




- Soft x-rays: forward scattering

  *D. Shapiro et al., PNAS **102**, 15343 (2005)*

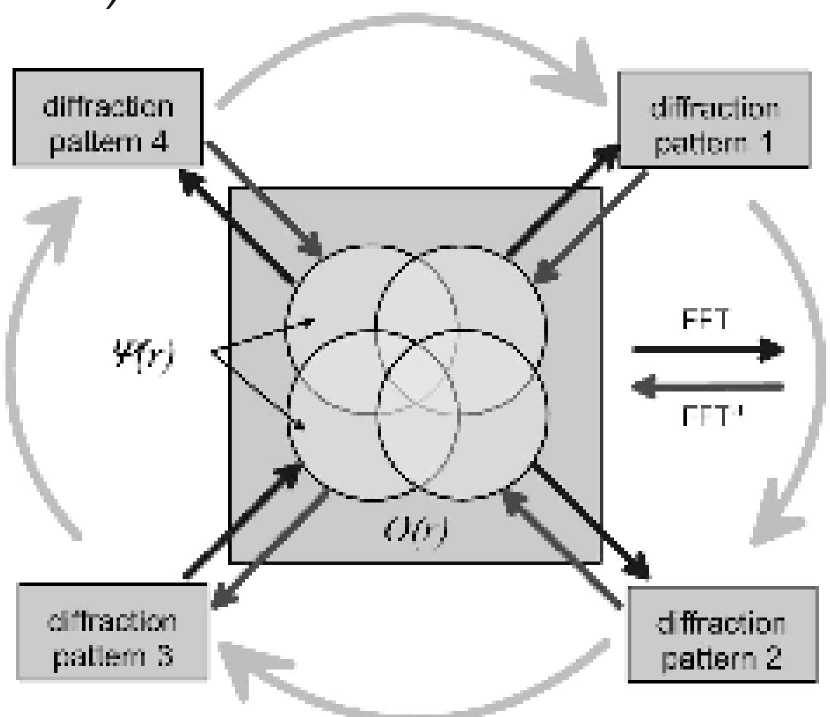


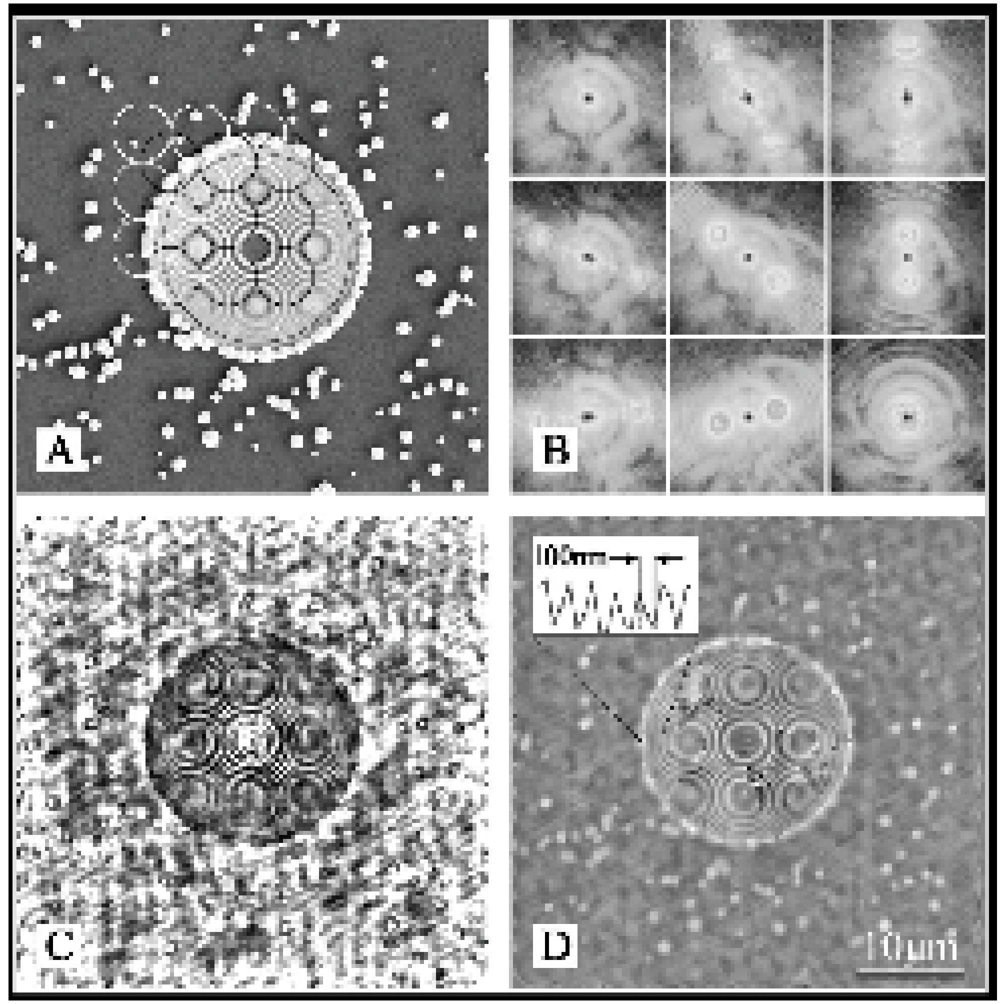


Coherent Imaging - Ptychography

- Record multiple speckle patterns from spatially overlapping regions
- Improves convergence, field of view

J. Rodenburg et al., PRL ***98****, 034801 (2007)*

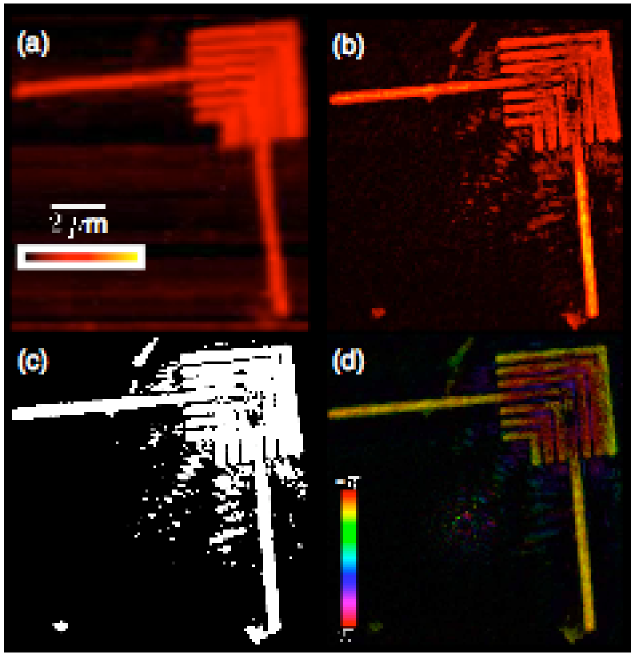




Coherent Imaging - Curved Wavefronts

- Using focusing to produce curved wavefront illumination improves algorithm convergence

*G. Williams et al., PRL **97**, 025506 (2006)*



Summary

- This is a very exciting time for nanoscale materials science
- The dramatically improving capabilities for hard x-ray imaging promise to have a strong impact
- Nanoprobe beamline capabilities include:
  - Scanning probe microscopy, with fluorescence and diffraction contrast
  - Nanoprobe will be great for coherence techniques: diffraction imaging, ptychography, XPCS
  - Stroboscopic time-resolved measurements
  - Polarization control e.g. to image magnetism